

Patent
Docket No.: 55416US002

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: Yaoqi J. Liu,
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Andrew T. Ruff, Brian L. Koster and
David K. Mortenson

Serial No.: 09/590,924

Group Art Unit: 1773

Filed: June 9, 2000

Examiner: Vivian Chen

For: **WRINKLE RESISTANT INFRARED REFLECTING FILM
AND NON-PLANAR LAMINATE ARTICLES MADE
THEREFROM**

Commissioner for Patents
Washington, D.C. 20231

AMENDMENT

CERTIFICATE OF FACSIMILE TRANSMISSION UNDER 37 C.F.R. 1.8: I hereby
certify that this correspondence is being sent by facsimile to the telephone number shown
below, addressed to the Commissioner for Patents, Washington, D.C. 20231, on the below
indicated date.

Facsimile Number: 703-872-9310

Date: 26 March 2002 By: David R. Cleveland

Dear Sir:

This paper is in response to the Office Action mailed December 26, 2001. Please
note that the application has a new attorney docket number (viz., 55416US002). Please
amend the application as follows:

Serial No. 09/590,924Docket No. 55416US002**In the Written Description:**

Make amendments to the following paragraph in the Written Description, as shown below in clean form and as shown in marked form in the attached document entitled "Marked copy of March 26, 2002 amendments to paragraphs of the Written Description in USSN 09/590,924 (55416US002)":

At page 1, lines 12-20:

A1
The laminate may also include at least one functional layer engineered to enhance the performance of the vehicle window. One important functional layer reduces entry of infrared radiation into the vehicle cabin. Infrared rejecting functional layers are typically made of metallized or dyed polymer film constructions that reflect or absorb unwanted solar radiation. When used in a windshield, the composite laminate structure should transmit at least about 70% of the light in the wavelength region sensitive to the human eye, typically from about 380 to about 700 nanometers (nm), and reject solar radiation outside the visible portion of the spectrum. When used in other glazing structures, such as side or rear windows, there are typically no limits on the level of visible transmission.

At page 2, lines 15-23:

A2
To enhance vehicle aerodynamics and improve outward visibility, vehicular window shapes are not planar, and increasingly include severe angles and complex curves. When the laminate 10 is placed between complex curved glass sheets and laminated with a nip roll process, or heated to bond the PVB to the glass, the functional layer 12 cannot perfectly conform to the complex curvatures, especially when the glass sheets are large. Wrinkles, folds and pleats can form in the functional layer, and, when the functional layer is metallized, cracks can form in the metallized layer 16 during nip rolling, which creates an optical defect in the safety glazing. As a result, only small size laminates with no curvature or a small one-dimensional curvature can currently be manufactured using a nip roll process.

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At page 4, lines 11-27:

A3

For example, by selecting the layer thicknesses to reflect near infrared light, and positioning the reflective bandedge within the infrared region such that even at grazing angles of incidence the reflectance band does not shift into the visible region of the spectrum, an infrared mirror can be made that is transparent in the visible region of the spectrum, even at high angles of incidence. The infrared (IR) reflecting films described in U.S. Patent Nos. 5,882,774 and 6,049,419, each incorporated herein by reference, control the amount of solar energy that pass through them, preferably without significantly decreasing the intensity or changing the color of light sensed by the human eye at any angle. The materials in the layers, the thicknesses of the layers, and the indices of refraction of the layers are selected to reflect infrared radiation within the wavelength range of about 700 nm to about 2000 nm, while transmitting visible light. The film has an average reflectivity of at least 50% over a band at least 100 nm wide in the infrared region of the spectrum. These films have been applied to substantially flat substrates to form laminates. However, when applied to a non-planar substrate, wrinkles form in the film, so the films have not been used in laminates with severely curved or compound curved substrates. The wrinkles are a particular problem in laminates that should be substantially optically clear, such as, for example, laminates intended for use in vehicular windshields.

At page 8, lines 9-18:

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To reflect over a wide band, the various layers in the film preferably have varying relative thicknesses, referred to herein as a layer thickness gradient, which are selected to achieve the desired bandwidth of reflection. In one embodiment, the layer thickness gradient may be linear, in which the thickness of the layer pairs increases at a constant rate across the thickness of the film, so that each layer pair is a certain percent thicker than the thickness of the previous layer pair. In another embodiment, the layer thickness may decrease, then increase, then decrease again from one major surface of the film to the other, or may have an alternate layer thickness distribution designed to increase the sharpness of one or both bandedges, as described in U.S. Patent No. 6,157,490, which is incorporated herein by reference.

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AS In an alternate embodiment, IR reflecting films including more than two distinguishable polymers can be made. This extends the reflecting band further into the IR to increase the amount of IR reflection while minimizing color in the visible region of the spectrum, which results from reflection due to higher order harmonics. Examples of such films include those described in U.S. Patent No. RE 34,605, incorporated herein by reference. The RE '605 patent describes a multilayer optical interference film including three diverse substantially transparent polymeric materials, A, B, and C and having a repeating unit of ABCB. The layers have an optical thickness of between about 90 nm to about 450 nm, and each of the polymeric materials has a different index of refraction, n_i .

At page 10, lines 4-10:

ALC Another useful film design is described in U.S. Patent No. 6,207,260, which is incorporated herein by reference. Optical films and other optical bodies are described which exhibit a first order reflection band for at least one polarization of electromagnetic radiation in a first region of the spectrum while suppressing at least the second, and preferably also at least the third, higher order harmonics of the first reflection band, while the percent reflection of the first order harmonic remains essentially constant, or increases, as a function of angle of incidence.

At page 13, line 20 through page 14, line 7:

AM A polyester useful in the solar rejection films of the present invention is polyethylene naphthalate (PEN), which can be made, for example, by reaction of naphthalene dicarboxylic acid with ethylene glycol. Polyethylene 2,6-naphthalate (PEN) is frequently chosen as a first polymer. PEN has a large positive stress optical coefficient, retains birefringence effectively after stretching, and has little or no absorbance within the visible range. PEN also has a large index of refraction in the isotropic state. Its refractive index for polarized incident light of 550 nm wavelength increases when the plane of polarization is parallel to the stretch direction from about 1.64 to as high as about 1.9. Increasing molecular orientation increases the birefringence of PEN. The molecular orientation may be increased by stretching the material to greater stretch ratios and holding other stretching conditions fixed. Other semicrystalline naphthalene dicarboxylic

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polyesters suitable as first polymers include, for example, polybutylene 2,6-Naphthalate (PBN), polyethylene terephthalate (PET), and copolymers thereof. Non-polyester polymers are also useful in creating polarizer films. For example, polyether imides can be used with polyesters, such as PEN and coPEN, to generate a multilayer reflective mirror. Other polyester/non-polyester combinations, such as polyethylene terephthalate and polyethylene (e.g., those available under the trade designation Engage 8200 from Dow Chemical Corp., Midland, MI), can be used. Suitable first polymers are described, for example, in Published PCT Application Nos. WO 99/36248, WO 99/36262 and WO 01/38907, and in U.S. Patent No. 6,268,961, which are incorporated herein by reference.

At page 15, line 1 through page 16, line 6:

A8

Preferred second polymers are homopolymers of polymethylmethacrylate (PMMA), such as those available from Ineos Acrylics, Inc., Wilmington, DE, under the trade designations CP71 and CP80, or polyethyl methacrylate (PEMA), which has a lower glass transition temperature than PMMA. Additional preferred second polymers include copolymers of PMMA (coPMMA), such as a coPMMA made from 75 wt% methylmethacrylate (MMA) monomers and 25 wt% ethyl acrylate (EA) monomers, (available from Ineos Acrylics, Inc., under the trade designation PERSPEXTM CP63), a coPMMA formed with MMA comonomer units and *n*-butyl methacrylate (nBMA) comonomer units, or a blend of PMMA and poly (vinylidene fluoride) (PVDF) such as that available from Solvay Polymers, Inc., Houston, TX under the trade designation SOLEFTM 1008. Yet other preferred second polymers include polyolefin copolymers such as poly (ethylene-co-octene) (PE-PO) available from Dow-Dupont Elastomers under the trade designation ENGAGETM 8200, poly (propylene-co-ethylene) (PPPE) available from Fina Oil and Chemical Co., Dallas, TX, under the trade designation Z9470, and a copolymer of atactic polypropylene (aPP) and isotactic polypropylene (iPP) available from Huntsman Chemical Corp., Salt Lake City, UT, under the trade designation REFLEXTM W111. Second optical layers can also be made from a functionalized polyolefin, such as linear low density polyethylene-g-maleic anhydride (LLDPE-g-MA) such as that available from E.I. duPont de Nemours & Co., Inc., Wilmington, DE, under the trade designation BYNELTM 4105.

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Particularly preferred combinations of first/second polymers for optical layers in IR reflective mirrors include PEN/PMMA, PET/PMMA or PET/coPMMA, PEN/ECDELTM, PET/ECDELTM, PEN/sPS, PET/sPS, PEN/coPET, PEN/PETG, and PEW/THVTM. ECDELTM is a trade designation for a copolyester ether elastomer available from Eastman Chemical Company (Kingsport, TN). THVTM is a trade designation for a fluoropolymer commercially available from Minnesota Mining and Manufacturing Company (3M), St. Paul, MN. PMMA refers to polymethyl methacrylate, coPET refers to a copolymer or blend based upon terephthalic acid (as described above), and PETG refers to a copolymer of PET employing a second glycol (usually cyclohexanedimethanol). sPS refers to syndiotactic polystyrene.

For mirror films, a match of the refractive indices of the first polymer and second polymer in the direction normal to the film plane is preferred, because it provides constant reflectance with respect to the angle of incident light (that is, there is no Brewster's angle). For example, at a specific wavelength, the in-plane refractive indices might be 1.76 for biaxially oriented PEN, while the film plane-normal refractive index might fall to 1.49. When PMMA is used as the second polymer in the multilayer construction, its refractive index at the same wavelength, in all three directions, might be 1.495. Another example is the PET/ECDELTM system, in which the analogous indices might be 1.66 and 1.51 for PET, while the isotropic index of ECDEL elastomer might be 1.52.

At page 18, line 13 through page 19, line 20:

A9

Various functional layers or coatings can be added to the films and optical devices of the present invention to alter or improve their physical or chemical properties, particularly along the surface of the film or device. Such layers or coatings may include, for example, low friction coatings or slip particles to make the film easier to handle during the manufacturing process; particles to add diffusion properties to the multilayer optical film or to prevent wet-out or Newton's rings when the multilayer optical film is placed next to another film or surface; adhesives such as pressure sensitive adhesives and hot melt adhesives, adhesion promoters, primers and low adhesion backside materials for use when the film is to be used in adhesive roll form. The functional layers or coatings may also include shatter resistant, anti-intrusion, or puncture-tear resistant films and coatings, for example, the functional layers described in commonly assigned Published PCT

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Application No. WO 01/96115 entitled GLAZING ELEMENT AND LAMINATE FOR USE IN THE SAME, incorporated herein by reference. Additional functional layers or coatings may include vibration-damping film layers such as those described in WO 98/26927 and U.S. Patent No. 5,773,102, barrier layers to provide protection or to alter the transmissive properties of the film or device towards liquids, such as water or organic solvents, or gases, such as oxygen, water vapor or carbon dioxide; and/or substrates and support layers designed to improve the mechanical integrity or strength of the film or device. These functional components may be incorporated into one or more skin layers, or they may be applied as a separate film or coating.

For some applications, it may be desirable to alter the appearance and/or performance of the multilayer optical film by coloring the film, such as by laminating a dyed film to the multilayer optical film, applying a pigmented coating to the surface of the film, or including a dye or pigment in one or more of the materials used to make the film, such as in the skin layer. The dye or pigment typically absorbs in one or more selected regions of the spectrum, including portions of the infrared, ultraviolet, and/or visible spectrum. The dye or pigment can be used to compliment the properties of the film, particularly where the film transmits some frequencies while reflecting others. A particularly useful pigmented layer that can be combined with the multilayer optical film of the present invention is described in Published PCT Application No. WO 01/58989, which is incorporated herein by reference. This film may be laminated, extrusion coated or coextruded as a skin layer on the multilayer film. The pigment loading level may be varied between about 0.01 and about 1.0% by weight to vary visible light transmission from about 10 to about 90%. In practice, the pigment loading level is selected such that when the pigmented film layer is combined with the multilayer optical film, the percent visible transmission is reduced to about 80-85% of the multilayer optical film's normal value, T_{vis} , to the legal limit of about 70-75% measured at an angle normal to a laminate construction. This improves the overall shading coefficient of the multilayer film construction.

4-11
At page 21, lines 4-23:

AIO

The above IR rejecting multilayer optical films can be made according to U.S. Serial No. 09/220724, which is incorporated herein by reference. These films solve the

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problems of cracking that are typically seen when metallized solar rejection films are nip roll laminated to glazings having compound curvature. The films may wrinkle during nip roll lamination, however, especially when applied to large laminates and/or glazings with severe compound curvatures. In another aspect of the present invention, the process conditions can be controlled to produce a film with improved shrinkage properties, resulting in a film that does not wrinkle during the nip roll lamination process.

A11

At page 21, line 12:

Process for Making Wrinkle-Free IR Film

A12

At page 22, lines 1-14:

If the non-planar substrate to which the film is to be laminated has a specific shape or curvature, to reduce wrinkling the shrinkage of the film may be individually controlled in each in-plane direction. If the curvatures along two principal axes of a region of the substrate are not equal, to laminate the film to that region without wrinkles, the shrinkage of the film may be controlled such that it differs in each in-plane direction. The in-plane direction of the film with the greater shrinkage should be aligned with the dimension of the substrate having the greater curvature (i.e., a smaller radius of curvature).

For example, to achieve substantially wrinkle-free lamination to a non-planar, curved or compound curved substrate, for a PEN or PET based film the shrinkage is greater than about 0.4% in both in-plane directions, preferably greater than about 0.7% in at least one in-plane direction, and more preferably greater than about 1% in at least one in-plane direction. The shrinkage should be maintained at a minimum to reduce edge delamination. This phenomenon is called a "pull-in." So, the shrinkage is preferably less than about 3% in each in-plane direction, more preferably less than about 2.5% in each in-plane direction.

A13

At page 22, line 28 through page 23, line 11:

For example, typical heatset conditions for PEN based multilayer IR mirror films intended for lamination to a planar substrate are about 480°F. To control shrinkage and provide a film suitable for wrinkle-free lamination to a non-planar substrate, the heat set temperature for a PEN based multilayer IR mirror film should be reduced to about 390°F

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to about 400°F, preferably about 395°F, for about 10 seconds. The heat set temperature for a PET based multilayer IR mirror film suitable for lamination to a non-planar substrate should be about 440°F to 470°F, preferably about 455°F to about 465°F. for about 10 seconds.

In addition to the heatset temperature and residence time, another parameter that affects film shrinkage is toe-in. Toe-in is defined as the decrease in rail spacing in the tenter heatset zone measured relative to a maximum rail setting. For example, if maximum width of the film is 67 inches at the end of the stretch zone, and film width in the heatset zone is 65 inches, then the toe-in is 2 inches. The first rail in the heatset zone is preferably set between the maximum rail setting and the rest of the heatset zone rail settings to ensure a smooth transition. However, tenter rail configuration and tenter width may vary widely, and ideal rail settings must be determined experimentally in each case.

At page 23, line 26 through page 24, line 2:

AI4

To control shrinkage and provide a film suitable for wrinkle-free lamination to a non-planar substrate, the toe-in should be adjusted depending on the polymer used. For a PEN based multilayer IR mirror film, the toe-in should be about 0 to 1.5 inches, preferably about 0.5 to 1 inches at the preferred heatset temperature, film width and heatset zone residence times mentioned above. For a PET based multilayer IR mirror film, the toe-in should be about 0 to 2 inches, preferably about 0 to 1.5 inches at the preferred conditions described above.

At page 25, lines 7-9:

AI5

If the multilayer IR film is laminated into a vehicular safety glazing laminate, the laminated structure will be substantially wrinkle-free, preferably optically clear, to the naked eye from a distance of less than about 1 meter, preferably less than about 0.5 meter.

At page 25, lines 17-29:

AI6

To ensure a substantially wrinkle-free lamination of the multilayer IR mirror film in a non-planar glazing article for a vehicle, such as a safety glazing laminate, the heating/cooling procedures and conditions in the lamination process should be carefully controlled. The temperatures used depend on the T_g of the polymeric components of the

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film and the viscosity and adhesion level of the mechanical energy absorbing layers, such as PVB, polyurethane (PUR) and SURLYN™, to the film. The preferred mechanical energy-absorbing layer is PVB. Preferably, some level of adhesion should be established between the PVB, the film and the non-planar glazing sheet(s) before the film shrinks during the pre-heating and de-airing process. However, the PVB should preferably not become sufficiently soft to flow before the film shrinks. The PVB should tack the edges and hold the IR film in position while the film shrinks and forms to the shape of the laminate. The laminate should be cooled after autoclaving at a controlled rate to avoid possible wrinkling due to the recovery of thermal expansion of the IR film and possible delamination on the edges.

A17

At page 26, lines 12-19:

To bond the PVB and IR film layers or the pre-laminate 110 or 140 to the glazing sheets 130, 132, the layers of PVB and IR film or the pre-laminate and the glazing sheets are assembled by placing them atop one another to form a unitary laminate structure 134. The layers of the laminate 134 are then bonded together and air is removed from the construction. During this step it is preferable to heat the laminate construction in an oven to a temperature below the Tg of the dominant polymer in the IR film 112. This allows the PVB layers 118, 120 to build some adhesion with the film 112 or the glass 130, 132 while the film 112 shrinks to form to the shape of the laminate.

A18

At page 28, line 21 through page 29, line 7:

The unprimed multilayer IR film samples were placed in glass/PVB/film/PVB/glass windshield laminates with compound curvatures. The DW 1224 windshield was intended for use in a model 1999 minivan manufactured by Daimler-Chrysler Corporation, Auburn Hills, MI, and has dimensions of 43 inches (109 cm) by 60 inches (152 cm). The TD direction of the film corresponded to the height of the windshield. Lamination trials were conducted using a vacuum de-airing process. Each laminate was prepared in a clean room. The laminates were wrapped with Ace bandages and then placed in a plastic bag. Once the bag was sealed, the laminate was de-aired by placing the bag under a vacuum of about 25 inches Hg at room temperature for minimum of 15 minutes. The laminates were then heat tacked in an oven at 220°F for about 45

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minutes. After removal from the oven, the edges were sealed with a glycol ether sealant available from Dow Chemical, Midland, MI under the trade designation DOWANOL™, and the laminates were placed in an autoclave. The startup temperature of the autoclave was about 100°F, and within about 17 minutes, the temperature increased to about 275°F and remained at that temperature for about 15 minutes. While the temperature was increasing, the pressure in the autoclave was also increased from room pressure to about 200 psi within about 19 minutes and remained at that level. After heat soaking was completed, the temperature was reduced to about 100°F within about 10 to 15 minutes. At about the same time, the pressure was also reduced to atmospheric pressure.

At page 29, lines 10-14:

A19

The windshield lamination results in Table 1 demonstrate that the film samples made at a lower tenter heat set temperature, less than about 440°F, preferably about 410°F or less, increased shrinkage of the film and resulted in a laminate with fewer or no wrinkles. Wrinkle-free lamination may be achieved when shrinkage is at least about 0.7% in both the MD and TD, preferably greater than about 1%.

At page 29, line 16 through page 30, line 14:

A20

A multilayer IR mirror film with 224 microlayers was prepared. The layers alternated between a first polymer of coPEN (90%PEN/10%PET) from 3M and a second polymer of PMMA CP80 from Ineos Acrylics. The film also included coPEN skin layers on the exterior of the multilayer stack. The thickness of the skins and PBLs as a percentage of the total film construction prior to lamination is listed in Table 2. The film was orientated first in the MD direction in a length orienter with a stretch ratio of about 3.3:1 and then in the TD direction in a tenter with a stretch ratio of about 4.0:1. The temperatures in the tenter were: preheat zones 275°F, stretch zones 280°F and cooling zones 120°F. The residence time in the heatset zone was approximately 10 seconds. The heatset temperature and toe-in conditions are listed in Table 2 below, along with the shrinkage properties of the film samples. Selected, unprimed film samples from a designed experiment were laminated into a windshield (DW1218) intended for use in a 1999 Taurus automobile manufactured by Ford Motor Company, Dearborn, MI. The windshield had compound curvatures and dimensions of 40 inches (102 cm) by 63 inches (160 cm).

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Sample were cleaned and prepared under standard production conditions. Two layers of 15 mil (0.038 cm) Dupont B14 PVB were used with IR film sandwiched in between and with one piece of glass on each outer side. After the entire construction was laminated and trimmed, a ring seal gasket was placed around the edge of the windshield and a vacuum was applied to the construction to remove air. While vacuum was applied, the laminate entered a warming oven for about eleven minutes at a temperature of about 212°F. After leaving the warming oven, the ring seal was removed and the samples were placed onto a holding rack.

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Fax Cover Sheet

26 March 2002

To: Examiner Vivian Chen From: David R. Cleveland
Group Art Unit 1773 Our Ref: 101-55416US002
Re: Serial No. 09/590,924 Direct Phone: 651-251-2250
Fax: 703-872-9310 Pages (including cover sheet): 35
Phone: _____ Confirmation: Yes _____ No X

Documents attached:

1. Amendment Transmittal Letter (in duplicate)
2. Amendment (32 pp)

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PATENT

Docket No.

55416US002

Amendment Transmittal Letter

Commissioner for Patents
Washington, D.C. 20231

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David K. Mortenson

Application No. 09/590,924

Group Art Unit: 1773

Filed: June 9, 2000

Examiner: Vivian Chen

Title: WRINKLE RESISTANT INFRARED REFLECTING FILM AND NON-PLANAR LAMINATE
ARTICLES MADE THEREFROM

- ☒ Enclosed is an Amendment (including marked pages showing changes) in the above-identified application.
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Claims As Amended						
	(2) Claims Remaining After Amendment		(4) Highest No. Previously Paid For	(5) Present Extra	(6) Rate	(7) Additional Fee
Total Claims	54	Minus	** 54	= 0	X \$18.	= \$0
Independent Claims	10	Minus	*** 10	=	X \$84.	= \$0
Additional fee for filing one or more multiple dependent claims, if no such fee has been previously paid					\$260.	\$0
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Registration Number	Telephone Number
29,524	651-251-2250
Date	
March 26, 2002	

Respectfully submitted,

Signature

Print Name

David R. Cleveland

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